PATENT ABSTRACTS OF JAPAN

(11) Publication number: 09-111165 (43) Date of publication of application: 28,04.1997

(51) Int, Cl. C09D 11/00 B41J 2/01 B41M 5/00

C09D 11/08

(21) Application number: 07-295998 (71) Applicant: CANON INC

(22) Date of filing: 20, 10, 1995 (72) Inventor: NOGUCHI HIROMICHI

MIYAGAWA MASASHI TAKENOUCHI MASANORI SHIMOMURA MASAKO

(54) INK AND INK TET RECORDING METHOD USING THE SAME

(57) Abstract:

PROBLEM TO BE SOLVED: To prepare an ink which can realize high-quality recordingthe formation of a high-definition color image and an improvement in reliability by incorporating a water-soluble dyewaterat least one compound selected from groups A and B of compds. and at least one compound selected from a group C of compds.

SOLUTION: A water-soluble dyewaterand a group A consisting of compds. (A-1 to A-9: nine compds. such as ethylene glycol monor-butyl etherwhich have high compatibility with water and an alkyl group through an ether bond in the molecular end) in an amt. of 6 to 12wt. based on the total amt. of the inkat least one compd. selected from a group B consisting of compds. (B-1 to B-3: three compds. such as surfactants based on ethylene oxide adduct to acetylene glycolwhich can impart rapid penetrability into plain paper) in an amt. of 0.3 to 3.0wt. based on the total amt. of the inkand at least one compd. selected from a group C consisting of compds. [two or more compds. represented by the formula (wherein (k) is 3 to 50; (m) is 3 to 25(n) is 3 to 25(m + m) is 6 to 50) in an amt. of 0.5 to 5wt. are incorporated so that the surface tension of the ink is regulated to not less than 30dyne/cm with the viscosity being modified

following general formula

CLAIMS

[Claim(s)]

[Claim 1]An ink composition which at least one sort of compounds selected from water soluble dyewaterthe following compound group Aand the compound group B and at least one sort of compounds chosen from the following compound group C are contained and are 30 or more dyne/cm of surface tensionand 5 or less centipoise of viscosity.

Compound group A:, A-1:. Ethylene glycol mono-n-butyl ether A-2:. Ethylene-glycol-monophenyl-ether A-3:. Ethylene glycol monoisobutyl ether A-4:. Diethylene-glycol mono-n-butyl ether A-5:. Diethylene-glycol monohexyl ether A-6:. Diethylene-glycol monoisobutyl ether A-6:. Diethylene-glycol monopropyl ether A-9: An ethylene oxide addition type surface-active agent B-2:ethylene oxide propylene oxide ethylene oxide type of an ethylene oxide addition compound group B:B-1:acetylene glycol of benzyl alcohol. (BURJRO nick type) surface-active agent B-3: — ethylene oxide addition compound group C:C-1: of higher alcohol — a compound expressed with a

It is k= 3-50. (k in a formula is 3-50m is 3-25m is 3-25and m+m is 6-50.)

C-2: A compound expressed with a following general formula

(It is k= 20-50 in a formulam is 10-25n is 10-25it is n+m=20-50and p is 2-5.)

[Claim 2] The ink composition according to claim 1 in which a compound of the compound group A occupies 6 to 12 weight % in the ink whole quantity. [Claim 3] The ink composition according to claim 1 in which a compound of the compound group B occupies 0.3 to 3.0 weight % in the ink whole quantity.

[Claim 4] The ink composition according to claim 1 in which a compound of the compound group C occupies 0.5 to 5 weight % in the ink whole quantity.

[Claim 5] The ink composition of four colors using yellowcyanogenmagentaand water soluble dye of each color of black according to claim 1 is usedAn ink jet recording method performing color recording using an ink-jet recording device which forms and carries out

the regurgitation of the droplet adjusted by the range of these ink whose volume of one droplet is 3-50 pico litersrespectively.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the ink jet recording method which carries and records the ink composition (only henceforth ink) and it which reconcile a high definition color picture and reliability.

[0002]

[Description of the Prior Art]With an ink-jet printeran ink tank is separated from the meaning which reduces the fall of a running costand waste with a print head in recent years recording equipment of the type for which only an ink tank is exchangeable is desired and such a product is already put in practical use in part.

[0003]Howeverby making it the recording equipment for which only an ink tank is exchanged has shown that the following problems may occur. That isalthough air naturally exists in the nozzle or the ink passage at the time of exchange of an ink tankwhen filled up there with new inkthe fault that air (bubble) will remain all over a tank may occur. In the ink jet recording method made especially into the driving force of droplet formation of a pressure pulsethe bubble which fixes and stagnates in ink has a possibility of causing a serious obstacle in subsequent droplet formation.

[0004]Development of the high-density print head for color ink jet recorders progressedand it has been [for / high-definition record] ready as a device. Howeverdynamical SUBJECT that the regurgitation of the droplet (the range of 3-50 pico liters) smaller than former is stably carried out to the ink for high-definition record with high frequencySUBJECT of the reliability that to control on a higher level on equipment is demanded has arisen without not getting ink clogged and ink stagnating in a nozzle surface.

[0005]Even if it performs the functional design which turned the device design to high definition—ization even if to many above SUBJECTmany above SUBJECT is unsolvable at all only by designing the material of ink only on the extension wire from the former. In order to satisfy the various characteristics required of the ink side to be used in order to solve many of these SUBJECTit is necessary to perform skillfully

combination of the ink component which bears various functions. Specifically selection and combination of many materials such as prevention from blinding of ink (evaporation reduction) a solvent which maintains ink to hypoviscosity a solvent which has solvent power to the color in inkand foam suppressorare very important. [0006]

[Problem to be solved by the invention] Therefore within a color ink jet recorder it is hard to produce obstacles — air is incorporated in foaming of inkor ink — therefore it is stabilized to a nozzle and the purpose of this invention can supply ink to it. The perviousness to the recording form of ink and the balance of a blot are adjusted the regurgitation characteristic is stablestable liquid particles can be formed also in small droplet formation and it is hard to get it clogged and is in providing the new ink which enables high-definition record. There is another purpose of this invention in providing the color ink jet record method excellent also in reliability while being able to form a high definition color picture.

[0007]

[Means for solving problem] The above-mentioned purpose is attained by the following this inventions. Namelyat least one sort of compounds in which this invention was chosen from water soluble dyewaterthe following compound group Aand the compound group BAnd it is a color ink jet record method which contains at least one sort of compounds chosen from the following compound group Cand uses the ink which is 30 or more dyne/cm of surface tensionand 5 or less centipoise of viscosityand this ink. Compound-group A:A-1: Ethylene glycol monor n-butyl ether (adhesion tension-34.3)

A-2: Ethylene-glycol-monophenyl-ether. (Adhesion tension-31.9) A-

3:ethylene glycol monoisobutyl ether (adhesion tension-32.5) A-4: Diethylene-glycol mono- n-butyl ether (adhesion tension-32.3) A-5:diethylene-glycol monohexyl ether. (Adhesion tension-31.7) mono- [A-6:diethylene-glycol] — isobutyl ether (adhesion tension-32.5) A-7:triethylene glycol n-butyl ether (adhesion tension-24.5) A-8:dipropyleneglycol monopropyl ether. (Adhesion tension-24.0) A-9: -- the ethylene oxide addition (adhesion tension-20.0) of benzyl alcohol — in additionthe unit of adhesion force is dyne/cm.
[0008]The ethylene-oxide addition type surface-active-agent B-

2:ethylene-oxide propylene-oxide ethylene-oxide type (BURURO nick type) surface-active agent B-3 of a compound-group B:B-L:acetylene glycol: The ethylene oxide addition of higher alcohol [0009]Compound-group C:C-1: The compound expressed with a following general formula

It is k=3-50. (k in a formula is 3-50m is 3-25n is 3-25and m+m is 6-50.)

C-2: The compound expressed with a following general formula

(It is k=20-50 in a formulam is 10-25n is 10-25it is n+m=20-50 and p is 2-5.)

[0010] It is possible according to this invention to be hard to produce obstacles — air is incorporated in foaming of ink or ink — therefore for it to be stabilized to a nozzle and to supply ink to it within a color ink jet recorder. The perviousness to the recording form of ink and the balance of a blot are adjusted and the regurgitation characteristic is stable. Stable liquid particles can be formed also in small droplet formation and it is hard to get it clogged and while being able to form the new ink which enables high-definition record and a high definition color picture the color ink jet record method excellent also in reliability can be provided.

[0011]

[Mode for carrying out the invention] Nexta desirable embodiment is mentioned and this invention is explained still in detail. In presentation the iok of this invention Water soluble dyewaterthe above-mentioned compound group A. At least one sort of compounds selected from the above-mentioned compound groups B and at least one sort of compounds chosen from the above-mentioned compound group C are contained it is characterized by being 30 or more dyne/cm of surface tensionand 5 or less centipoise of viscosity in physical properties.

 $[0012] \\ Bereafter it$ explains from the main characterizing portion of this invention.

1. The solution which each compound of the compound group A compound group A has high compatibility with waterand contains this compound 6weight % or more has the high-speed perviousness corresponding to color recordingits volatility is lowits viscosity is loward its flash point is also highand its color solubility is also good. Howeverif these compounds are contained 20weight % or morewhen using paper as a recording mediumosmosis on the back side of paper becomes remarkableand is not preferred. Using the necessity for the compound group A in this inventionsufficient content is the 6 weight % or more 12 or less weight % of range preferably.

[0013]Each compound of the compound group A is a substance which have an alkyl group through the other bond of the molecular terminaland viscosity becomes lowerd has fixed hydrophobicity and has surface

activity ability though it is a solvent. Such character is a reason suitable for high-speed osmosis of ink. The comparison data of the adhesion tension (a dynamic angle of contact and meaning are the same) by the dynamic wettability testing machine of each compound of the compound group A was shown above. Measurement shows numerically the adhesion tension of 5 seconds after [when contacting the compound of 0.5 weight % (the remainder is water) of colors which have added various glycol other containing the compound group A in small quantities for the purpose of only 10 weight % and discernment on paper on fixed conditions]. The wettability in a short time expresses the high thingso that it is a negative big value. According to this invention person's etc. measurementin the ink of this inventiondesirable various characteristics were acquired by using the substance in which the adhesion tension not more than -20 dyne/cm is shown under this measuring condition.

[0014]2. the ethylene oxide addition of a compound group B**B-1:acctvlene glvcol -- the substance used here is the compound to which the acetylene glycol was made to carry out 3.5-20-mol additional coupling of the ethylene oxide on an average. Specifically as a compound marketedSAFI Norian 440 (3.5-mol addition)SAFI Norian 465 (10-mol addition) (the above is the Nissin Chemical make) ASECHI Norian EH (10mol addition) (Product made from Kawaken fine)etc. are mentioned, 5 mol15 mola 20-mol additionetc, can give high perviousness. **B-2: an ethylene oxide propylene oxide ethylene oxide type (BURURO nick type) surface-active agent -- these are known as a nonionic surface-active agent. The substance in which the hydrophobicity of the range of 4-8 has strong HLB is used suitably for this invention in these. [0015]**8-3: the ethylene oxide addition of higher alcohol - these are known as a nonionic surface-active agent. The substance in which the hydrophobicity of the range of 4-8 has strong HLB is used suitably for this invention in these, ink [as opposed to a regular paper by choosing either of each compound of B-1 of a more than B-2 and B-3 and using it in 0.5 to 1.0 weight % of the range preferably 0.3 to 3.0 weight % in the ink of this invention] -- quick perviousness can be obtained. [0016]3. Each compound of the compound group C compound group C has the operation which gives foam inhibition nature and antifoam to inkalthough the compatibility with water is not so high. This compound is effective when especially being used together with the compound group A. It is for the substance of the compound group A improving the solubility of the substance of the compound group C into inkand demonstrating the foam

inhibition operation effectively, ** General structure of the compound

[0017]Molecule both ends are propylene glycol and there is the feature of the substance structure of C-I compound in the middle of a molecule having ethylene glycol structure. While this compound has fixed hydrophobicity in an aqueous medium by taking such a structureit is thought that the hydrophobic atom group (propylene glycol) of both sides lowers surface tensionand gives foam inhibition nature in the surface in addition.

[OO18]As a silicone series defoaming agent which has silicone architecture in a molecular terminalalthough many substances are developed they will be in the state where emulsification dispersion was carried out in the drainage system mediumand will serve as a heterogeneous system. Although such a silicone series defoaming agent is excellent in the function as a defoaming agentit is lacking in long-term stabilityand surface tension serves as a low value of 30 or less dyne/cmand it is hard to form it into a droplet particleand its bolding power of a meniscus is low unsuitable. In the above-mentioned general formula (C-1) substancein the range of 4-10and a molecular weightHLB is liquefied oligomer of the range of 400-5000 a desirable compound is k= 3-50m is 3-25 and is n+m=6-50.

[0019] ** General structure of the compound of C-2

It comes out. It is the substance which contained the alkyl silicone diol unit in order that the compound C-2 of the above-mentioned general formula might improve feam inhibition/antifeam to C-1The range of 4-10 and a melecular weight are liquefied eligomer of the range of 400-5000HLB is k= 20-50m is 10-25n is 10-25it is n+m=20-50and p is 2-5. These compounds are not new substancesare blended as an additive agent for ink as a propylene glycel ether system defeaming agentand are used. In the ink of this inventioneither C-1 or C-2 is generally preferably used in 10.1-2.0 weight % 0.5 to 5.0weight %although it changes with target ink-medium systems. Although a concrete addition changes with target ink-medium systemsit is added in the range which compatibility permits.

[0020] In the ink of this invention the compound belonging to these compound groups C has surface activity ability and lessens winning of the air at the time of the droplet formation stable by its foam inhibition / defeaming operation and ink restoration. Since the compound group C is dissolving thoroughly into inkthe characteristic thing cannot reduce the surface tension of ink itself remarkably and can maintain the surface

tension of 30 or more dyne/cm which is one of the conditions of the ink of this invention.

[0021]4. The color material in color material (color) this invention is water soluble dye. If the color used by this invention is water soluble dye for drainage system ink jet inkany publicly known color can be used for it. In the ink of this inventiondesirable water soluble dye is illustrated below. These colors are the substance groups selected in order it is clearand the solubility in the medium of ink is stableto excel in the color tone at the time of recording on paper and to perform waterproof good record. Although the following colors are mentionedspecificallythis invention is not limited to these colors. The water-soluble groups in an illustration color may be a respectively free statealkaline metal saltammonium saltand organic amine salt. [0022] Marcota dve (1)

[0023]Magenta dye (2)

[0024] Magenta dve (3)

[0025] Magenta dve (4)

[0026] Yellow dye (1)

[0027]Yellow dye (2)

[0028]Yellow dye (3)

[0029] Yellow dye (4)

[0030]Cyan dye (1)

[0031]Cyan dye (2)

```
[0032]Cyan dye (3)
Triphenylmethane series ABL-9
[0033]Black dye (1)
[0034]Black dye (2)
[0035]Black dye (3)
[0036]Black dye (4)
```

Although the above-mentioned color is a compound with the fitness of ink for ink jet recordingit is needless to say that a suitable color can be used out of water soluble dyesuch as acid dye and a direct coloralso except these choosing. The amount of these water soluble dye used is a quantity which occupies about 0.5 to 5.0 weight % in the ink whole quantity.

[0038]5. Explain the liquid medium of inknext the liquid medium of ink. the liquid medium of the ink of this invention — water — although it may be independentit is desirable when adding the high water miscibility solvent of moistness in water considers it as more reliable ink. Specifically Ethylene glycola diethylene glycoltriethylene glycolTripropylene glycolglycerin124-butanetrio1126-bexanetrio11 and 25-pentanetrio112-butanedio113-butanedio114-butanedio10imethyl sulfoxidediacetone alcoholglycerin monoallyl etherPropylene glycola butylene glycolthe polyethylene glycol 300thiodiglycolN-methyl-2-pyrrolidone2-pyrrolidonegamma-butyrolactone13-dimethyl-2-imidazolidinonesulfolanetrimethylolpropaneTrimethylolethaneneopentyl glycolethylene glycol monomethyl etherEthylene glycol monocthyl etherethylene glycol monomispropyl etherEthylene glycol monocthyl ethertriethylene glycol monomethyl ethertriethylene glycol monomethyl ethertriethylene glycol monomethyl ethertriethylene glycol monomethyl ethertriethylene glycol monomethyl

etherpropylene glycol monomethyl etherDipropylene glycol monomethyl etherScrew beta-hydroxyethyl sulfonescrew beta-hydroxyethyl ureaureaacetonylacetonepentaerythritoll4-cyclohexanedioletc, are mentioned. As for these water soluble organic solventsit is preferred to use it at a rate of about five to 50 weight section per water 100 weight section.

[0039]6. As for pH of the ink of pH adjuster this inventionadjusting to basicity from neutrality is more preferred from a viewpoint of the stability of ink than from the character of the water soluble dye to be used. As a pH adjuster for

that specifically Ethanolamine diethanolamine triethanolamine N-methylethanolamine N-ethyldiethanolamine 2: amino-2-methyl propanol 2: ethyl-2-amino-13-propanediol 2-(2-aminoe thyl) ethanolamine Tris (hydroxymethyl) aminomethane ammonia a glycine Organic bases such as a glycylglycine histidine L-lysine L-arginine piperidine morpholine and screw beta-bydroxyethyl urea; bases such as sodium hydroxidea potassium hydrate and lithium hydroxideare mentioned. It is also possible to give the pll stability of ink by a buffer for pl. Also in these bases especially in order to acquire the preservation stability of inkand the jetting stability of the ink on an ink-jet recording device alcohol

amine is preferred. [0040] 7. Explain preparation of inknext preparation of the ink of this invention. The ink of this invention is prepared by 35 - 45 dyne/cmandas for 30 or more dyne/cm of surface tension5 or less centipoises of viscosity are preferably prepared by two to 4 centipoise. In order to acquire these physical properties the substance chosen from the compound group B 15 or less weight % 6weight % or more in the substance chosen from the compound group A must be blended in 5 or less weight % of the range 0.5weight % or more. In the range of the material composition of inkand the range of physical properties priority must be given to the above-mentioned property rangeand it must opt for combination. Although the ink of this invention is used suitably for an ink-jet recording deviceit cannot be overemphasized that it is not necessarily limited to this focurseand is applied also to other mono color ink jet recording methods and other record methods.

[0041]8. Explain a color ink jet record methodnext the color ink jet record method of this invention. The ink of this invention is designed the best for an on-demand type high-definition color ink jet printer. The fundamental composition of such a color ink jet printer in the multi-nozzle recording head provided with the piezoelectric element or the heater elementWore than nozzle density 180dpi (dot per inch) is

equipment which records above the droplet volume 10 - 20pl (pico-litter) and the storage density of 300 lpi (line per inch). When ink is exbaustedit is suitable also for the recording equipment of the type for which only an ink tank can be exchanged.

[0042]

[Working example]Nextan embodiment and a comparative example are given and this invention is explained still more concretely. The part in an embodiment and a comparative example or % is a weight reference as long as there is no notice especially.

Ammonium salt three-copy and compound-A-9 of enbodiment l and black dye (1) Ten copies (2 mol of ethylene oxide addition of benzyl alcohol)

- Compound C-1 4 copy (n+m=16k= 12the molecular weight 1450
- [about]HLB=7.3 (calculated value))

The 50% isopropyl alcohol solution of a ** liquefied compound

-1 copy of ethylene glycol ten-copy and tris(hydroxymethyl) aminomethane (pH adjuster) and water 72 copies[0043]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand pH 8.3surface tension 42.0 dyne/cmand black ink GEI-HB of this example with a viscosity of 2.2 cps were obtained. Each ink of cyanogen (GEI-1C) magenta (GEI-1M) and yellow (GEI-1Y) was similarly produced except having replaced the color with the following by the above-mentioned formula.

- 1 copy of diethylene-glycol 13-copy and ammonium sulfate (pll adjuster) and water 71.2 copies[0046]Agitation mixing was improved the abovementioned formulait filtered under pressure with a 0.2-micrometer membrane filterand pll 8.5surface tension 36.0 dyne/cmand cyan color ink GEI-2C of this example with a viscosity of 2.5 cps were obtained. Wagenta (GEI-2M)yellow (GEI-2Y)and each black (GEI-2B) ink were similarly produced except having replaced the color with the following

[0047]Ammonium salt 2.8-copy and compound-A-7 of embodiment 3 and magenta dye (1) Six copies (triethylene glycol n-butyl ether) — The compound C-2 Four copies (n+m=20p= 5k= 2050% ME TANORU solution of molecular weight about 2500 compound)

- 1 copy of diethylene-glycol 15-copy and triethanolamine (pH adjuster) and water 72.2 copies[0048]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand pH 9.0surface tension 40.0 dyne/cmand magenta color ink GEI-3M of this example with a viscosity of 2.1 cps were obtained. Each ink of black (GEI-3B) cyanogen (GEI-3C) and yellow (GEI-3Y) was similarly produced except having replaced a color with the following by the above-mentioned formula.

[0049]Ammonium salt 2.8-copycompound-A-2 (SAFI Norian 465) 1.5-copyand the compound C-2 of embodiment_4 and yellow dye (2) Five copies (n+m=50p= 3k= 3020% ME TANORU solution of molecular weight about 4500 compound)

- I copy of glycerin eight-copydiethylene-glycol 15-copyand triethanolamine (pH adjuster) and water 72.2 copies[0050]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand pH 9.2surface tension 35.0 dyne/cmand yellow color ink GEI-4Y of this example with a viscosity of 2.7 cps were obtained. Each ink of black (GEI-4B)magenta (GEI-4A) and cyanogen (GEI-4C) was similarly produced except having replaced a color with the following by the above-mentioned formula.

[0051]Ammonium salt 2.8-copy and compound-A-8 of <u>embodiment 5</u> and black dye (2) Ten copies (dipropyleneglycol monopropyl ether)

- Compound B-2 Two copies (an ethylene oxide propylene oxide ethylene oxide type (BURURONI KKU type) surface-active agentHLB-Smolecular weight 2500 [about])

" Compound C-1 4 copy (n+m=30k= 25the molecular weight 2850

[about]HLB=7.7 (calculated value))
The 50% ethyl alcohol solution of *********

- 0.3 copy of diethylene-glycol five-copyglycerin ten-copyand lithium hydroxide (pH adjuster) and water 72.2 copies[0052]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand black ink GEI-5B of with pH 9.5 surface tension of 38.0 dynes/cm and a viscosity of 3.0 cps this example was obtained. Each ink of a cyan color (GEI-5C)magenta (GEI-5W) and yellow (GEI-5Y) was similarly produced except having replaced the color with the following by the above-mentioned formula.

[0053]Comparative example ink SF1-1B which improved perviousness with the surface-active agent of an <u>one or less-comparative example</u> formula was prepared.

- Ethylene oxide addition of ammonium salt three copy and and nonyl phenol of black dye (1) One copy (HLB=10Sannopuko make)
- 1 copy of urea 7.5-copydiethylene-glycol 12.5-copyand
- tris(hydroxymethyl) aminomethane (pH adjuster) and water Agitation mixing is improved 75 copies of substances of theselt filtered under pressure with a 0.2-micrometer membrane filterand pH 8.0surface tension 30.0 dyne/cmand black ink SFI-1B of the comparative example 1 with a viscosity of 2.5 cps were obtained.

[0054]Comparative example ink SFDF1-2B which added the silicone series defoaming agent of a <u>two or less-comparative example</u> formulaand planned foam inhibition was prepared.

- Ethylene oxide addition of ammonium salt three copy and and nonyl phenol of black dye (1) One copy (HLB=10Sannopuko make)
- 2 copies of copolymer [of I copy of urea 7.5 copydiethylene-glycol 12.5 copyand tris(hydroxymethyl) aminomethane (pH adjuster) and silicone dioland ethylene oxide] (defoaming agent)and water 73 copies of substances of these with a homogenizer. It agitated for 15 minutes at 100 rpmit filtered under pressure with a 0.2-micrometer membrane filterand pH 8.0surface tension 26.5 dyne/cmand black ink SFDFI-2B of the comparative example 2 with a viscosity of 2.6 cps were obtained. This ink is a solution which is in the state where the defoaming agent
- was emulsified and lacks in a transparent feeling a little.
 [0055]In black ink GEI-IB of comparative example 3 Embodiment (compound-A-9 (1 mol of ethylene oxide addition of benzyl alcohol) is reduced from

ten copies to four copiesInsteadblack ink CSFI-3B of the comparative example 3 was obtained completely like Embodiment 1 except having added the diethylene glycol. The viscosity of this ink was 44.0 dynos/cm in 2.55 cps and surface tension.

[0056][Printing examination] To each ink of Embodiments 1-5 and the comparative examples 1-3128 nozzlesThe following examinations were done using nozzle density 720dpihorizontal scanning and the vertical-scanning print density of 720 lpithe drive frequency of 8 kHzthe trial production bubble jet color printer that has the characteristic of average droplet volume 10pland the electrophotography copy paper (regular paper).

** The average value of droplet volume was measured with the equipment which can monitor the form at the time of the regurgitation from the nozzle of regurgitation characteristic-droplet formation droplet (volume).

(Valuation basis)

A: The main droplet of 10pl is breathing out about.

Droplet smaller than B:2 10pl is breathing out.

C: It does not become droplet but is breathing out with the state of a liquid column.

D: It is divided in much small droplets and is breathing out.

[0057]** All the ink of the ink tank of one ink-tank-replacement examination is exhaustedload with the new ink tank of the same colordraw in from the nozzle side with a recovery pumpand perform operation which fills up a head with new ink. Subsequentlysolid printing followed until it exhausted the ink was performed.

(Valuation basis)

A: It finished printing satisfactorily at all to normal number of sheets. B: Although the ink piece happened on the wayit was able to return to the normal state by restoring operation.

C: The ink piece often happened and pump sucking operation was performed frequently. Although many fine bubbles were looked at by the ink chamber in ex post facto observationthere was no damage to a heater.

D: The ink piece happenedprinting density faded and printing continuation was not able to be carried out in restoring operation after that. In expost facto observationmany bubbles fine in a liquid ink room were seenand damage to a heater was also seen.

[0058]** It recorded on the regular paper which described above the printability-drying text and the test pattern which allotted the mono color patchand drying time was examined.

(Valuation basis)

A: It is dry when discharged from the printer.

B: Dry in several seconds after being discharged.

C: Dry in tens of seconds after being discharged.

[0059]** The boundary part and character between the color patches of the printing quality obtained by the print quality-boundary blot "** printability-drying property" were observed and the state of the blot was investigated.

(Valuation basis)

A: Visuallythere is no boundary blot between colors.

B: The boundary blot has occurred a little between a yellow color and black.

C: The line of a character is stout and a boundary blot is conspicuous. Howeverin the blot examination of the ink of a comparative example each color ink of Embodiment 1 was used for color ink other than black. [0060] [Evaluation result] The evaluation result of each item of the above-mentioned printing examination is shown in Table 2. Table 2: Evaluation result of Embodiments 1-5 and comparative examples 1-3

[0061]

[Effect of the Invention] As explained aboveaccording to this inventionare hard to produce obstaclessuch as incorporating foaming or air of recording ink within recording equipmentherefore the stable supply for a nozzle is possible in order to adjust the perviousness to the recording form of inkand the balance of a blotto be able to perform stable liquid particle formation also in droplet formation stable [the regurgitation characteristic] and small to be hard to get it clogged and to enable high-definition record the record method using the new ink which reconciled the image quality and reliability of highly minute color ink jet equipmentand this ink is provided. By taking the composition of the ink of this inventionan inexpensive and good picture can be created now in an officeand it is useful to the large spread of digital printing technology.

[0062]By using the ink of this invention for an ink-jet recording device** Droplet formation where being divided in the small droplet called a splash was stabilized few can be performedAnd also when it records on ** regular paper from which regurgitation durability is acquiredosmosis fixing time is short. And the picture which has few unique boundary blots (bleeding) and in which color enhancement is goodand they are clear is acquired. When using the ink-jet recording device of the type for which only an ink tank is exchangeablethe effect of filling up with ink certainly [there are little incorporation of air

and foaming out of inkand] to an ink passage is acquired also at the time of ** ink tank replacement.

WRITTEN AMENDMENT

-----[Written

Amendment]

[Filing date]Heisei 8(1996) September 27

[Amendment 1]

[Document to be Amended]Description

[Item(s) to be Amended] Whole sentence

[Method of Amendment] Change

[Proposed Amendment]

[Document Name]Description

[Title of the Invention] Ink and a color ink jet record method using it [Claim(s)]

[Claim 1] Ink which at least one sort of compounds selected from water soluble dwewaterthe following compound group Aand the compound group B and at least one sort of compounds chosen from the following compound group C are containedand is 30 or more dyne/cm of surface tensionand 5 or less centipoise of viscosity.

The compound group A:

A-1: Ethylene glycol mono- n-butyl ether

A-2: Ethylene glycol monophenyl ether

A-3: Ethylene glycol monoisobutyl ether

A-4: Diethylene-glycol mono- n-butyl ether

A-5: Diethylene-glycol monohexyl ether

A-6: Diethylene-glycol monoisobutyl ether

A-7: Triethylene glycol n-butyl ether

A-8: Dipropyleneglycol monopropyl ether

A-9: An ethylene oxide addition of benzyl alcohol

The compound group B:

B-1: An ethylene oxide addition type surface-active agent of an acetylene glycol

B-2: An ethylene oxide propylene oxide ethylene oxide type (BURURO nick type) surface-active agent

B-3: An ethylene oxide addition of higher alcohol

The compound group C:

C-1: A compound expressed with a following general formula

(k in a formula is 3-50m is 3-25m is 3-25and n+m is 6-50.) C-2: A compound expressed with a following general formula

(1t is k= 20-50 in a formulam is 10-25n is 10-25it is n+m=20-50and p is 2-5.)

[Claim 2] The ink composition according to claim 1 in which a compound of the compound group A occupies 6 to 12 weight % in the ink whole quantity. [Claim 3] The ink composition according to claim 1 in which a compound of the compound group B occupies 0.3 to 3.0 weight % in the ink whole quantity.

[Claim 4]The ink composition according to claim 1 in which a compound of the compound group C occupies 0.5 to 5 weight % in the ink whole quantity.

[Claim 5] The ink according to claim 1 in which water soluble dye occupies 0.5 to 5.0 weight % in the ink whole quantity.

[Claim 6] The ink according to claim 1 containing a water soluble organic solvent.

[Claim 7]The ink according to claim 1 whose water soluble dye is magenta dye.

[Claim 8] The ink according to claim 1 whose water soluble due is yellow due.

[Claim 9] The ink according to claim 1 whose water soluble dye is cyan dye.

[Claim 10] The ink according to claim 1 whose water soluble dye is black dye.

[Claim II] In a color ink jet record method which makes these ink an ink droplet with an inkjet methodrespectively and performs color recording by these ink droplets using magentayellow cyanogenand each black color ink color ink jet record method wherein ink of each above-mentioned color is the ink according to claim 1.

[Claim 12] These ink is made into an ink droplet adjusted with an inkjet method by range whose volume per drop is 3-50 pico litersrespectively using magentayellowcyanogenand each black color inkA color ink jet record method characterized by ink of each above-mentioned color being the ink according to claim I in a color ink jet record method which performs color recording by these ink droplets.

[Detailed Description of the Invention]

[Field of the Invention] This invention relates to the color ink jet record method which carries and records the ink composition (only henceforth ink) and it which reconcile a high definition color picture and reliability.

[0002]

[Description of the Prior Art]With an ink-jet printeran ink tank is separated from the meaning which reduces the fall of a running costand waste with a print head in recent years recording equipment of the type for which only an iok tank is exchangeable is desired and such a product is already put in practical use in part.

[0003]Howeverby making it the recording equipment for which only an ink tank is exchanged has shown that the following problems may occur. That is although air naturally exists in the nozzle or the ink passage at the time of exchange of an ink tankwhen filled up there with new inkthe fault that air (bubble) will remain all over a tank may occur. In the ink jet recording method made especially into the driving force of droplet formation of a pressure pulsethe bubble which fixes and stagnates in ink has a possibility of causing a serious obstacle in subsequent droplet formation.

[0004]Development of the high-density print head for color ink jet recorders progressedand it has been [for / high-definition record] ready as a device. Howeverdynamical SUBJECT that the regurgitation of the droplet (the range of 3-50 pico liters) smaller than former is stably carried out to the ink for high-definition record with high frequencySUBJECT of the reliability that to control on a higher level on equipment is demanded has arisen without not getting ink clogged and ink stagnating in a nozzle surface.

[0005]Even if it performs the functional design which turned the device design to high definition-ization even if to many above SUBJECTmany above SUBJECT is unsolvable at all only by designing the material of ink only on the extension wire from the former. In order to satisfy the various characteristics required of the ink side to be used in order to solve many of these SUBJECTit is necessary to perform skillfully combination of the ink component which bears various functions. Specifically selection and combination of many materials such as prevention from blinding of ink (evaporation reduction) a solvent which maintains ink to hypoviscosity a solvent which has solvent power to the color in inkand foam suppressorare very important.

[Problem to be solved by the invention] Thereforewithin a color ink jet recorderit is hard to produce obstacles — air is incorporated in foaming of inkor ink — therefore it is stabilized to a nozzle and the purpose of this invention can supply ink to it. The perviousness to the recording form of ink and the balance of a blot are adjusted the regurgitation characteristic is stablestable liquid particles can be formed also in small droplet formation and it is hard to get it cloggedand is in providing the new ink which enables high-definition record. There is another purpose of this invention in providing the color ink jet record method excellent also in reliability while being able to form a high definition color picture.

[0007]

[Means for solving problem] The above-mentioned purpose is attained by the following this inventions. Namelyat least one sort of compounds in which this invention was chosen from water soluble dyewaterthe following compound group Aand the compound group BAnd it is a color ink jet record method which contains at least one sort of compounds chosen from the following compound group Cand uses the ink which is 30 or more dyne/cm of surface tensionand 5 or less centipoise of viscosityand this ink. Compound group A:

A-1: Ethylene glycol mono- n-butyl ether (adhesion tension-34.3)

A-2: Ethylene glycol monophenyl ether (adhesion tension-31.9)

A-3: Ethylene glycol monoisobutyl ether (adhesion tension-32.5)

A-4: Diethylene-glycol mono- n-butyl ether (adhesion tension-32.3)

A-5: Diethylene-glycol monohexyl ether (adhesion tension-31.7)

A-6: Diethylene-glycol monoisobutyl ether (adhesion tension-32.5)

A-7: Triethylene glycol n-butyl ether (adhesion tension-24.5)

A-8: Dipropyleneglycol monopropyl ether (adhesion tension-24,0)

A-9: The ethylene oxide addition of benzyl alcohol (adhesion tension- $20,0)\,$

The unit of adhesion tension is dyne/cm.

[0008]Compound group B:

B-1: The ethylene oxide addition type surface-active agent of an acetylene glycol

B-2: Ethylene oxide propylene oxide ethylene oxide type (BURURO nick type) surface-active agent

B-3: The ethylene oxide addition of higher alcohol

[0009]Compound group C:

C-1: The compound expressed with a following general formula

(k in a formula is 3-50m is 3-25n is 3-25and n+m is 6-50.) C-2: The compound expressed with a following general formula

(It is k= 20--50 in a formulam is 10--25n is 10--25it is n+m=-20--50 and p is 2--5.)

[0010] It is possible according to this invention to be hard to produce obstacles — air is incorporated in foaming of ink or ink — therefore for it to be stabilized to a nozzle and to supply ink to it within a color ink jet recorder. The perviousness to the recording form of ink and the balance of a blot are adjusted and the regurgitation characteristic is stable. Stable liquid particles can be formed also in small droplet formation and it is hard to get it clogged and while being able to form the new ink which enables high-definition record and a high definition color picture the color ink jet record method excellent also in reliability can be provided.

[0011]

[Mode for carrying out the invention] Nexta desirable embodiment is mentioned and this invention is explained still in detail. In presentation the ink of this invention Water soluble dyewaterthe abovementioned compound group A. At least one sort of compounds selected from the above-mentioned compound groups B and at least one sort of compounds chosen from the above-mentioned compound group C are contained it is characterized by being 30 or more dyne/cm of surface tensionand 5 or less centipoise of viscosity in physical properties.
[0012] Hereafterit explains from the main characterizing portion of this

[0012]Hereafterit explains from the main characterizing portion of this invention.

1. Compound group A

The solution which each compound of the compound group A has high compatibility with waterand contains this compound 6weight % or more has the high-speed perviousness corresponding to color recordingits volatility is lowits viscosity is lowand its flash point is also highand its color solubility is also good. Howeverif these compounds are contained 20weight % or morewhen using paper as a recording mediumosmosis on the back side of paper becomes remarkable and is not preferred. Using the necessity for the compound group A in this inventionsufficient content is the 6 weight % or more 12 or less weight % of range preferably.

[0013] Each compound of the compound group A is a substance which have an

alkyl group through the ether bond of the molecular terminaland viscosity becomes lowand has fixed hydrophobicityand has surface activity ability though it is a solvent. Such character is a reason suitable for high-speed osmosis of ink. The comparison data of the adhesion tension (a dynamic angle of contact and meaning are the same) by the dynamic wettability testing machine of each compound of the compound group A was shown above. Measurement shows numerically the adhesion tension of 5 seconds after I when contacting the compound of 0.5 weight % (the remainder is water) of colors which have added various glycol ether containing the compound group A in small quantities for the purpose of only 10 weight % and discernment on paper on fixed conditions]. The wettability in a short time expresses the high thingso that it is a negative big value. According to this invention person's etc measurementin the ink of this inventiondesirable various characteristics were acquired by using the substance in which the adhesion tension not more than -20 dyne/cm is shown under this measuring condition.

[0014]2. Compound group B

**B-1: The ethylene oxide addition of an acetylene glycol
The substance used here is the compound to which the acetylene glycol
was made to carry out 3.5-20-mol additional coupling of the ethylene
oxide on an average. Specifically as a compound marketedSAFI Norian 440
(3.5-mol addition)SAFI Norian 465 (10-mol addition) (the above is the
Nissin Chemical make)ASECHI Norian EH (10-mol addition) (Product made
from Kawaken fine)etc. are mentioned. 5 moll5 mola 20-mol additionetc.
can give high perviousness.

**B-2: Ethylene oxide propylene oxide ethylene oxide type (BURURO nick type) surface-active agent

These are known as a nonionic surface-active agent. The substance in which the hydrophobicity of the range of 4--8 has strong HLB is used suitably for this invention in these.

[0015]***B-3: The ethylene oxide addition of higher alcohol These are known as a nonionic surface-active agent. The substance in which the hydrophobicity of the range of 4-8 has strong HLB is used suitably for this invention in these. The quick perviousness of the ink to a regular paper can be obtained by choosing either of each compound of B-1 of a more thanB-2and B-3and using it in 0.5 to 1.0 weight % of the range preferably 0.3 to 3.0 weight % in the ink of this invention. [0016]3. Compound group C

Each compound of the compound group C has the operation which gives foam inhibition nature and antifoam to inkalthough the compatibility with

water is not so high. This compound is effective when especially being used together with the compound group A. It is for the substance of the compound group A improving the solubility of the substance of the compound group C into inkand demonstrating the foam inhibition operation effectively.

** General structure of the compound of C-1

[0017]Molecule both ends are propylene glycol and there is the feature of the substance structure of C-1 compound in the middle of a molecule having ethylene glycol structure. While this compound has fixed hydrophobicity in an aqueous medium by taking such a structureit is thought that the hydrophobic atom group (propylene glycol) of both sides lowers surface tensionand gives foam inhibition nature in the surface in addition.

addition. [0018] As a silicone series defoaming agent which has silicone architecture in a molecular terminalalthough many substances are developed they will be in the state where emulsification dispersion was carried out in the drainage system medium and will serve as a heterogeneous system. Although such a silicone series defoaming agent is excellent in the function as a defoaming agent is lacking in long-term stability and surface tension serves as a low value of 30 or less dyne/cmand it is hard to form it into a droplet particleand its holding power of a meniscus is low unsuitable. In the above-mentioned general formula (C-1) substancein the range of 4-10and a molecular weightHLB is liquefied oligomer of the range of 400-5000a desirable compound is k= 3-50m is 3-25and n is 3-25 and is n+m=6-50.

[0019]** General structure of the compound of C-2

It comes out. It is the substance which contained the alkyl silicone diol unit in order that the compound C-2 of the above-mentioned general formula might improve foam inhibition/antifoam to C-1The range of 4-10 and a molecular weight are liquefied oligomer of the range of 400-5000HLB is k= 20-50m is 10-25n is 10-25it is n+m=20-50and p is 2-5. These compounds are not new substances are blended as an additive agent for ink as a propylene glycol other system defoaming agentand are used. In the ink of this inventioneither C-1 or C-2 is generally preferably used in 0.1 to 2.0 weight % of the range 0.5 to 5.0 weight % although it

changes with target ink-medium systems. Although a concrete addition changes with target ink-medium systemsit is added in the range which compatibility permits.

[0020] In the ink of this invention the compound belonging to these compound groups C has surface activity ability and lessens winning of the air at the time of the droplet formation stable by its foam inhibition / defeaming operation and ink restoration. Since the compound group C is dissolving thoroughly into inkthe characteristic thing cannot reduce the surface tension of ink itself remarkably and can maintain the surface tension of 30 or more dyne/cm which is one of the conditions of the ink of this invention.

[0021]4. Color material (color)

The color material in this invention is water soluble dye. If the color used by this invention is water soluble dye for drainage system ink jet inkany publicly known color can be used for it. In the ink of this inventiondesirable water soluble dye is illustrated below. These colors are the substance groups selected in order it is clearand the solubility in the medium of ink is stableto excel in the color tone at the time of recording on paper and to perform waterproof good record. Although the following colors are mentionedspecificallythis invention is not limited to these colors. The water—soluble groups in an illustration color may be a respectively free statealkaline metal saltammonium saltand organic amine salt.

[0022]Magenta dve (1)

[0023] Magenta dve (2)

[0024] Magenta dye (3)

[0025] Magenta dve (4)

[0026] Yellow dye (1)

```
[0027] Yellow dye (2)
[0028] Yellow dye (3)
[0029]Yellow dye (4)
[0030]Cyan dye (1)
[0031]Cyan dye (2)
[0032]Cyan dye (3)
Triphenylmethane series ABL-9
[0033]Black dye (1)
[0034]Black dye (2)
[0035]Black dye (3)
[0036]Black dye (4)
```

Although the above-mentioned color is a compound with the Fitness of the ink for ink jet recordingit is needless to say that a suitable color can be used out of water soluble dyesuch as acid dye and a direct coloralso except these choosing. The amount of these water soluble dye used is a quantity which occupies about 0.5 to 5.0 weight % in the ink whole quantity.

[0038]5, Liquid medium of ink

Next the liquid medium of ink is explained, the liquid medium of the ink of this invention - water - although it may be independentit is desirable when adding the water soluble organic solvent of the high water miscibility of moistness in water considers it as more reliable ink. Specifically Ethylene glycola diethylene glycoltriethylene glycolTripropylene glycolglycerin124-butanetriol126-hexametriol1 and 25pentanetriol12-butanediol13-butanediol14-butanediolDimethyl sulfoxidediacetone alcoholglycerin monoallyl etherPropylene glycola butylene glycolthe polyethylene glycol 300thiodiglycolN-methyl-2pyrrolidone2-pyrrolidonegamma-butyrolactone13-dimethy1-2imidazolidinonesulfolanetrimethylolpropaneTrimethylolethaneneopentyl glycolethylene glycol monomethyl etherEthylene glycol monoethyl etherethylene glycol monoisopropyl etherEthylene glycol monoallyl etherdiethylene glycol monomethyl etherdiethylene glycol monoethyl ethertriethylene glycol monomethyl ethertriethylene glycol monoethyl etherpropylene glycol monomethyl etherDipropylene glycol monomethyl etherscrew beta-hydroxyethyl sulfonescrew beta-hydroxyethyl ureaureaacetonylacetonepentaerythritoll4-cyclohexanedioletc. are mentioned. As for these water soluble organic solventsit is preferred to use it at a rate of about five to 50 weight section per water 100 weight section.

[0039]6. PH adjuster

As for pH of the ink of this inventionadjusting to basicity from neutrality is more preferred from a viewpoint of the stability of ink than from the character of the water soluble dye to be used. As a pH adjuster for thatspecificallyEthanolaminediethanolaminetriethanolamineN-methylethanolamineN-ethyldiethanolamine2-amino-2-methylpropanol2-ethyl-2-amino-13-propanediol2-(2-aminoethyl) thanolamineTris(hydroxymethyl)

aminomethaneammoniaa glycineOrganic basessuch as a glycylglycineOrstidineL-lysineL-argininepiperidinemorpholineand screw beta-hydroxyethyl urea; basessuch as sodium hydroxidea potassium hydrateand lithium hydroxideare mentioned. It is also possible to give the pH stability of ink by a buffer for pH. Also in these basesespecially in order to acquire the preservation stability of inkand the jetting stability of the ink on an ink-jet recording devicealcohol amine is preferred.

[0040]7. Preparation of ink

Nextpreparation of the ink of this invention is explained. The ink of this invention is prepared by 35 - 45 dyne/cmandas for 30 or more dyne/cm of surface tensions or less centipoises of viscosity are preferably prepared by two to 4 centipoises. In order to acquire these physical propertiesthe substance chosen from the compound group B 15 or less weight % 6weight % or more in the substance chosen from the compound group A must be blended in 5 or less weight % of the range 0.5weight % or more. In the range of the material composition of inkand the range of physical propertiespriority must be given to the abovementioned property rangeand it must opt for combination. Although the ink of this invention is used suitably for a color ink jet recorderit cannot be overemphasized that it is not necessarily limited to this of courseand is applied also to other mono color ink jet recording methods and other record methods.

[0041]8. Color ink jet record method

Nextthe color ink jet record method of this invention is explained. The ink of this invention is designed the best for an on-demand type high-definition color ink jet printer. The fundamental composition of such a color ink jet printerIn the multi-nozzle recording head provided with the piezoelectric element or the heater elementMore than nozzle density 180dpi (dot per inch) is equipment which records above the droplet volume 10 - 20pl (pico-litter) and the storage density of 300 lpi (line perinch). When ink is exhaustedit is suitable also for the recording equipment of the type for which only an ink tank can be exchanged. [0042]

[Working example] Nextan embodiment and a comparative example are given and this invention is explained still more concretely. The part in an embodiment and a comparative example or % is a weight reference as long as there is no notice especially.

Embodiment 1

- Ammonium salt of black dye (1) Three copies
- " Compound-A=9 Ten copies

(2 mol of ethylene oxide addition of benzyl alcohol)

- Compound C-1 Four copies

(n+m=16k=12the molecular weight 1450 [about]HLB=7.3 (calculated value))

The 50% isopropyl alcohol solution of a ** liquefied compound

- Ethylene glycol Ten copies
- One copy of tris(hydroxymethyl) aminomethane (pH adjuster)
- Water 72 copies

[0043]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand black ink GEI-IB of with pH 8.3 surface tension of 42.0 dynes/cm and a viscosity of 2.2 cps this example was obtained. Each ink of cyanogen (GEI-IC)magenta (GEI-IM) and yellow (GEI-IY) was similarly produced except having replaced the color with the following by the above-mentioned formula. [0044]

[0045]Embodiment 2

- Ammonium salt of cyan dye (2) 2.8 copies
- Compound-A-8 Eight copies

(Dipropyleneglycol monopropyl ether)

- Compound C-1 Four copies

(n+m=30k= 25the molecular weight 2850 [about]HLB=7.7 (calculated value))

The 50% ethyl alcohol solution of *******

- Diethylene glycol 13 copies
- One copy of ammonium sulfate (pH adjuster)
- Water 71.2 copies

[0046]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand pH 8.5 sourface tension 36.0 dyne/cmand cyan color ink GEI-2C of this example with a viscosity of 2.5 cps were obtained. Magenta (GEI-2M)yellow (GEI-2Y) and each black (GEI-2B) ink were similarly produced except having

replaced the color with the following by the above-mentioned formula.

[0047] Embodiment 3

- Ammonium salt of magenta dye (1) 2.8 copies
- Compound-A-7 Six copies

(Triethylene glycol n-butyl ether)

- Compound C-2 Four copies

(n+m=20p= 5k= 2050% ME of molecular weight about 2500 compound) TANORU solution

- Diethylene glycol 15 copies
- One copy of triethanolamine (pH adjuster)
- Water 72.2 copies

[0048]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand pH 9.0 surface tension 40.0 dyne/cmand magenta color ink GEI-3M of this example with a viscosity of 2.1 eps were obtained. Each ink of black (GEI-3B) cyanogen (GEI-3C) and yellow (GEI-3Y) was similarly produced except having replaced the color with the following by the above-mentioned formula.

[0049]Embodiment 4

- Ammonium salt of yellow dye (2) 2.8 copies
- Compound-A-2 (SAFI Norian 465) 1.5 copy
- The compound C-2 Five conies

(n+m=50p= 3k= 3020% ME of molecular weight about 4500 compound) A TANORU solution

- Glycerin Eight copies
- Diethylene glycol 15 copies
- One copy of triethanolamine (pH adjuster)
- Water 72.2 copies

[0050] Agitation mixing was improved the above-mentioned formulait

filtered under pressure with a 0.2-micrometer membrane filterand pH 9.2 surface tension 35.0 dyne/cmand yellow color ink GEI-4V of this example with a viscosity of 2.7 cps were obtained. Each ink of black (GEI-4B) magenta (GEI-4M) and cyanogen (GEI-4C) was similarly produced except having replaced the color with the following by the abovementioned formula.

[0051]Embodiment 5

- Ammonium salt of black dve (2) 2,8 copies
- Compound-A-8 Ten copies

(Dipropyleneglycol monopropyl ether)

- Compound B-2 Two copies

(Ethylene oxide propylene oxide ethylene oxide type (BURURONI))

A KKU type surface-active agentHLB=8the molecular weight 2500 [about]

- Compound C-1 Four copies

 $(n+m=30k=25the\ molecular\ weight\ 2850\ [\ about\]HLB=7.7\ (calculated\ value))$

The 50% ethyl alcohol solution of *******

- Diethylene glycol Five copies
- Glycerin Ten copies
- 0.3 copy of lithium hydroxide (pH adjuster)
- Water 72.2 copies

[0052]Agitation mixing was improved the above-mentioned formulait filtered under pressure with a 0.2-micrometer membrane filterand black ink GEI-5B of with pH 9.5 surface tension of 38.0 dynes/cm and a viscosity of 3.0 eps this example was obtained. Each ink of a cyan color (GEI-5C)magenta (GEI-5M) and yellow (GEI-5Y) was similarly produced except having replaced the color with the following by the above-mentioned formula.

[0053]Comparative example 1

Comparative example ink SFI-1B which improved perviousness with the surface-active agent of the following formulas was prepared.

Ammonium salt of black dye (1) Three copies

- Ethylene oxide addition of nonyl phenol One copy (HLB=10Sannopuko make)
- Urea 7.5 copies
- Diethylene glycol 12.5 copies
- One copy of tris(hydroxymethyl) aminomethane (pH adjuster)
- Water 75 copies

Agitation mixing was improved those substancesit filtered under pressure with a 0.2-micrometer membrane filterand pH 8.0surface tension 30.0 dyne/cmand black ink SFI-IB of the comparative example 1 with a viscosity of 2.5 cps were obtained.

[0054]Comparative example 2

Comparative example ink SFDF1-2B which added the silicone series defoaming agent of the following formulasand planned foam inhibition was prepared.

- Ammonium salt of black dye (1) Three copies
- Ethylene oxide addition of nonyl phenol One copy (MLB=10Sannopuko make)
- Urea 7.5 copies
- Diethylene glycol 12.5 copies
- One copy of tris(hydroxymethyl) aminomethane (pH adjuster)
- Two copies of copolymers (defoaming agent) of silicone diol and ethylene oxide
- Water 73 copies

With the homogenizerthese substances were agitated for 15 minutes at 100 rpmwere filtered under pressure with a 0.2-micrometer membrane filterand pll 8.0 surface tension 26.5 dyne/cmand black ink SFDFI-2B of the comparative example 2 with a viscosity of 2.6 cps were obtained. This ink is a solution which is in the state where the defoaming agent was emulsified and lacks in a transparent feeling a little.

[0055]Comparative example 3

In black ink GEI-1B of Embodiment Lcompound-A-9 (1 mol of ethylene oxide addition of benzyl alcohol) is reduced from ten copies to four copiesInsteadblack ink CSFI-3B of the comparative example 3 was obtained completely like Embodiment 1 except having added the diethylene glycol. The viscosity of this ink was 44.0 dynes/cm in 2.55 cps and surface tension.

[0056][Printing examination] To each ink of Embodiments 1-5 and the comparative examples 1-3128 nozzlesThe following examinations were done using nozzle density 720dpihorizontal scanning and the vertical-scanning print density of 720 lpithe drive frequency of 8 kHzthe trial production bubble iet color printer that has the characteristic of average droulet

volume 10pland the electrophotography copy paper (regular paper).

** Regurgitation characteristic-droplet formation

The average value of droplet volume was measured with the equipment which can monitor the form at the time of the regurgitation from the pozzle of droplet (volume).

(Valuation basis)

A: The main droplet of 10pl is breathing out about.

Droplet smaller than B:2 10pl is breathing out.

C: It does not become droplet but is breathing out with the state of a liquid column.

D: It is divided in much small droplets and is breathing out.

[0057] ** Ink-tank-replacement examination

All the ink of one ink tank is exhaustedand it loads with the new ink tank of the same colorand draws in from the nozzle side with a recovery pumpand operation which fills up a head with new ink is performed. Subsequentlysolid printing followed until it exhausted the ink was performed.

(Valuation basis)

A: It finished printing satisfactorily at all to normal number of sheets.

B: Although the ink piece happened on the wayit was able to return to

the normal state by restoring operation.

C: The ink piece often happened and pump sucking operation was performed frequently. Although many fine bubbles were looked at by the ink chamber

in ex post facto observation there was no damage to a heater.

D: The ink piece happened printing density faded and printing

that. In ex post facto observationmany bubbles fine in a liquid ink room were seenand damage to a heater was also seen.

[0058]** Printability-drying property

It recorded on the regular paper which described above the text and the test pattern which allotted the mono color patchand drying time was examined.

(Valuation basis)

A: It is dry when discharged from the printer.

B: Dry in several seconds after being discharged.

C: Dry in tens of seconds after being discharged.

[0059]** Print quality-boundary blot

The boundary part and character between the color patches of the printing quality obtained with "** printability-drying property" were observed and the state of the blot was investigated.

(Valuation basis)

- A: Visuallythere is no boundary blot between colors.
- B: The boundary blot has occurred a little between a yellow color and black.
- C: The line of a character is stout and a boundary blot is conspicuous. Howeverin the blot examination of the ink of a comparative exampleeach color ink of Embodiment I was used for color ink other than black. [0060] [Evaluation result] The evaluation result of each item of the above-montioned printing examination is shown in Table I.
- Table 1: Evaluation result of Embodiments 1-5 and comparative examples 1-3 $\,$

[0061]

[Effect of the Invention]As explained aboveaccording to this inventionare hard to produce obstaclessuch as incorporating foaming or air of ink within recording equipmenttherefore the stable supply for a nozzle is possibleln order to adjust the perviousness to the recording form of inkand the balance of a blotto be able to perform stable liquid particle formation also in droplet formation stable [the regurgitation characteristic] and smallto be hard to get it elogged and to enable high-definition recordThe record method using the new ink which reconciled the image quality and reliability of highly minute color ink jet equipmentand this ink is provided. By taking the composition of the ink of this inventionan inexpensive and good picture can be created now in an officeand it is useful to the large spread of digital printing technology.

- [0062]What the ink of this invention is used for an ink-jet recording device for
- *** Droplet formation where being divided in the small droplet called a splash was stabilized few can be performedand regurgitation durability is acquired
- ** When the picture with them which has few unique boundary blots (bleeding) is acquired and the ink-jet recording device of the type for which only an ink tank is exchangeable is used[short and osmosis fixing time and] [clear / color enhancement is goodand / also when it records on a regular paper 1
- ** Also at the time of ink tank replacementhe effect of filling up with ink certainly [there are little incorporation of air and foaming out of inkand] to an ink passage is acquired.